Phase I Test Results Joint Test Protocol for Cadmium Alternatives for High Strength Steel

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Report Documentation Page

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"High Strength Steel Joint Test Protocol for Validation of Alternatives to Low Hydrogen Embrittlement Cadmium for High Strength Steel Landing Gear and Component Applications" dated July 2003

3-phase Test Plan

- Phase I: Hydrogen Embrittlement and Re-embrittlement
- Phase II: General Properties, Adhesion, Corrosion, Lubricity, Repairability
- Phase III: Fatigue
- Phase I tests conducted at NAVAIR Pax River and Army Research Lab
- 7 primary and 4 repair coatings evaluated (including 3 baseline coatings)
- Coatings were nominated for evaluation by JCAT members

Coatings and Coaters

Coating Parameters

- 0.5 mil coating thickness requested
 - Zn based coatings tended to be thinner
 - Al based coatings tended to be thicker
- Post-plate bakeout (if applicable)
- Chromate conversion coated
- No underplate
- Surface prep included abrasive grit blast (except Sn-Zn)

Coatings and Coaters

Primary Coatings

- LHE Cadmium (baseline), Hill Air Force Base
- IVD Aluminum (baseline, unpeened), Hill Air Force Base
- Sputtered Aluminum, Marshall Laboratories
- Electroplated Aluminum, AlumiPlate Incorporated
- Dipsol IZ-C17 LHE Zn-Ni, Boeing St. Louis
- "Acidic" Zn-Ni, Boeing Seattle
- Sn-Zn, Dipsol of America

Repair Coatings

- Brush LHE Cadmium (SIFCO 2023)(baseline), Boeing St. Louis
- Brush Zn-Ni (SIFCO 4018), Boeing St. Louis
- Brush Sn-Zn (LDC 5030), Boeing St. Louis
- Spray Sermetel 249/273, Boeing St. Louis



- Acceptance Criteria: 4 of 4 specimens sustain 75% notch fracture strength (NFS) load for 200 hours without fracture; OR 1 of 4 specimens fracture in less than 200 hours and the remaining 3 sustain at least 1-hour at 90% NFS during subsequent incremental step loading.
- Specimens: ASTM F 519 Type 1a.1, 4340 alloy steel, HRC 51-53.
- All coatings passed this test except Sn-Zn. Failure analysis showed specimens had large intergranular fracture areas initiated at the notch surface, indicating embrittlement of the steel during processing.

Coating	<u>Fracture</u> <u>Time to</u>		Pass/Fail
	Strength (%)	Failure (hrs)	
LHE Cadmium	91.8	203	PASS
IVD Aluminum	98.4	203	PASS
Alumiplate	95.3	204	PASS
Sputtered Al	83.1	201	PASS
ZnNi (Boeing acidic)	93.1	203	PASS
LHE ZnNi (Dipsol IZ-C17)	92.0	203	PASS
SnZn	75.2	38	FAIL

Coating	Notch Tensile Strength (ksi)	Pass/Fail
Sputtered	359.8	FAIL
Aluminum	343.4	FAIL
Bare (avg. of 10 bars)	391.4	N/A

Criteria: NTS within 10 ksi of the average NTS of 10 bare specimens.

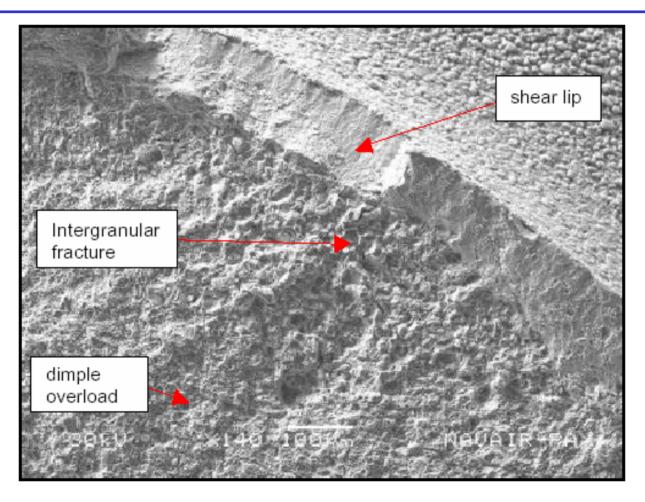
<u>Conclusion:</u> Sputtered Aluminum specimens detempered during processing. Atypical compared to previous experience.

<u>Coating</u>	Fracture Strength (%)	<u>Time to</u> <u>Failure (hrs)</u>	Pass/Fail
Brush Cadmium	91.7	203	PASS
Brush ZnNi	86.4	156	CLOSE
Brush SnZn	94.4	203	PASS
Sermetel 249/273	95.2	204	PASS

<u>Brush</u> <u>ZnNi</u>	<u>Fracture</u> Strength (%)	<u>Time to</u> <u>Failure (hrs)</u>
1	89.2	202
2	90.4	202
3	90.6	203
4	75.2	16.9

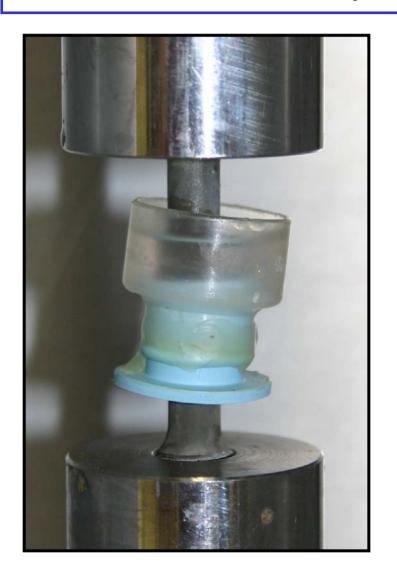
Failure Analysis:

- Bar #1: Fully ductile.
- Bar #4: Intergranular fracture (embrittlement) initiated below the surface.



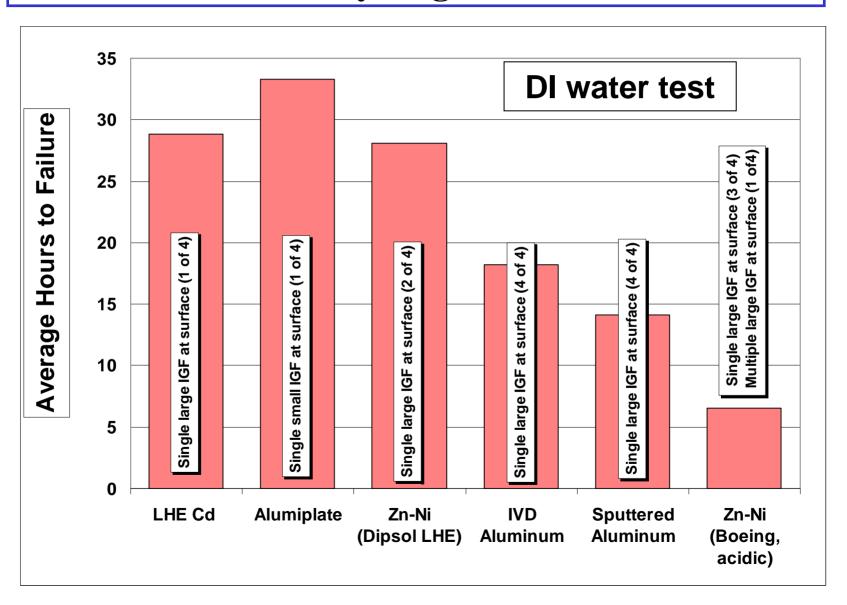
Brush Zn-Ni specimen #4, failed at 75.2% NFS @ 16.9 hrs.

- Acceptance Criteria: Average load and time to fracture greater than or equal to LHE Cd when tested in 1 mega ohm reagent water.
- **Specimens:** ASTM F 519 Type 1a.1, 4340 alloy steel, HRC 51-53.
- Loading profile: 45% NFS for 24 hrs, step 5% per hr until failure.
- Best performance: Alumiplate, LHE Cd, Dipsol IZ-C17 LHE ZnNi.
- IVD and Sputtered Aluminum provided significantly less protection from in-service re-embrittlement than LHE Cd.
- Boeing's "acidic" Zn-Ni provided the least protection.
- Other test fluids included 3:1 propylene glycol/Dlwater and ASTM D1141 synthetic sea water.



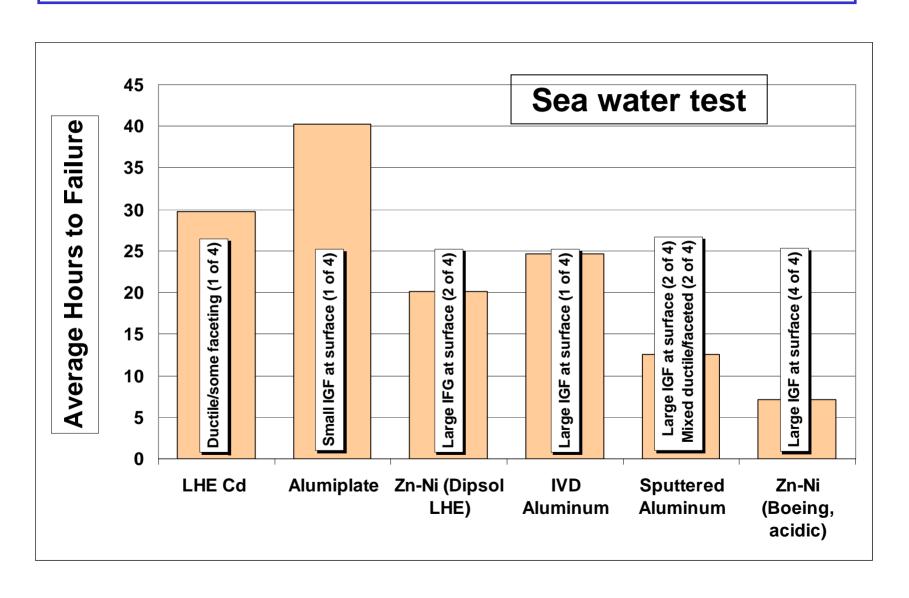
Test Setup

- All specimens loaded separately (no gang loading).
- Service fluids in cup isolated around notch, approximately 2-3 ml fluid in each cup.
- Fluids were not replenished during test – parafilm wrapped around grips prevent evaporation.



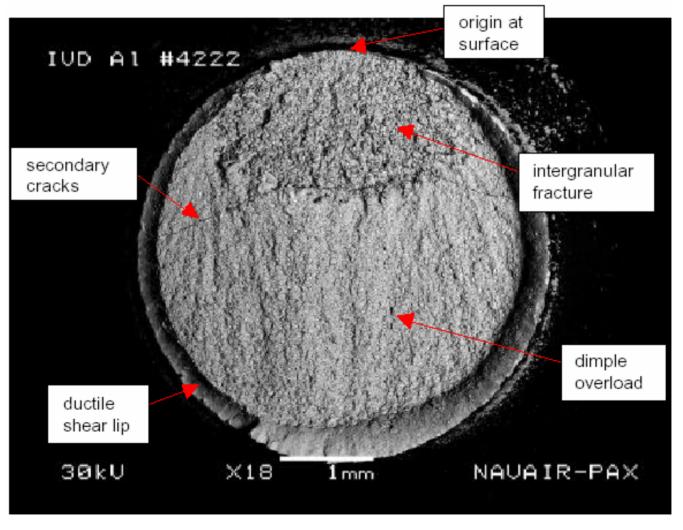
<u>Coating</u>	<u>Fracture</u> <u>Strength (%)</u>	<u>Load at</u> <u>Failure (lbs)</u>	Time to Failure (hrs)	Pass/Fail
LHE Cadmium	73.9	6958	28.8	N/A
IVD Aluminum	50.2	4720	18.2	N/A
Alumiplate	95.0	8940	33.3	PASS
Sputtered Al	47.7	4489	14.1	FAIL
LHE ZnNi (Dipsol IZ-C17)	70.2	6402	28.1	CLOSE
ZnNi (Boeing acidic)	46.4	4370	6.6	FAIL

DI water test

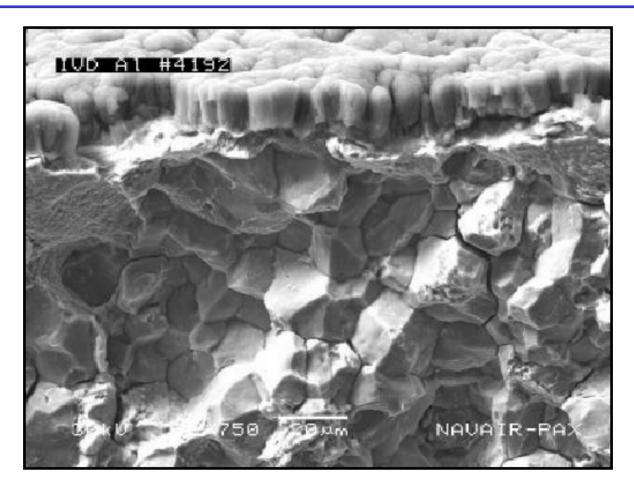


Coating	Fracture Strength (%)	<u>Load at</u> <u>Failure (lbs)</u>	<u>Time to</u> <u>Failure</u> (hrs)	Pass/Fail
LHE Cadmium	77.7	7316	29.7	N/A
IVD Aluminum	52.7	4956	24.6	N/A
Alumiplate	93.9	8841	40.3	PASS
Sputtered Al	49.0	4610	12.5	FAIL
LHE ZnNi (Dipsol IZ-C17)	57.6	5253	20.2	CLOSE
ZnNi (Boeing acidic)	46.4	4364	7.1	FAIL

Sea water test

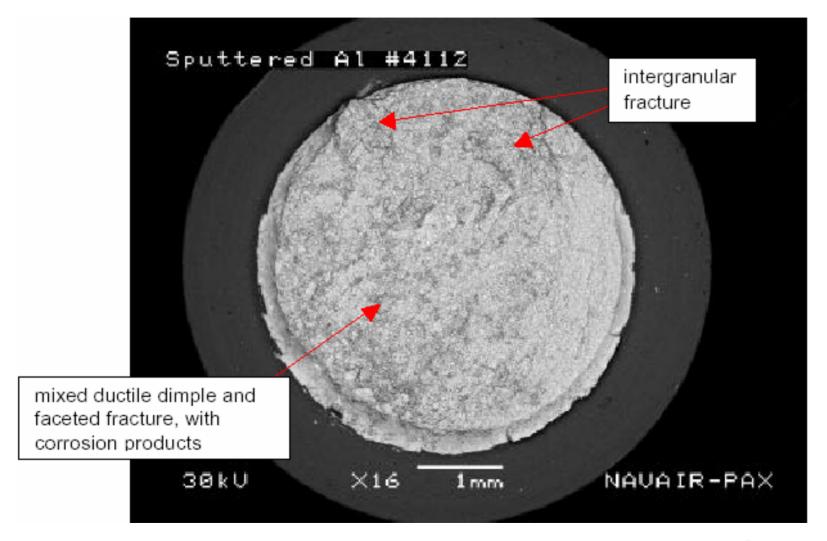


IVD Al tested in DI water, failed at 45% NFS @ 18 hrs.

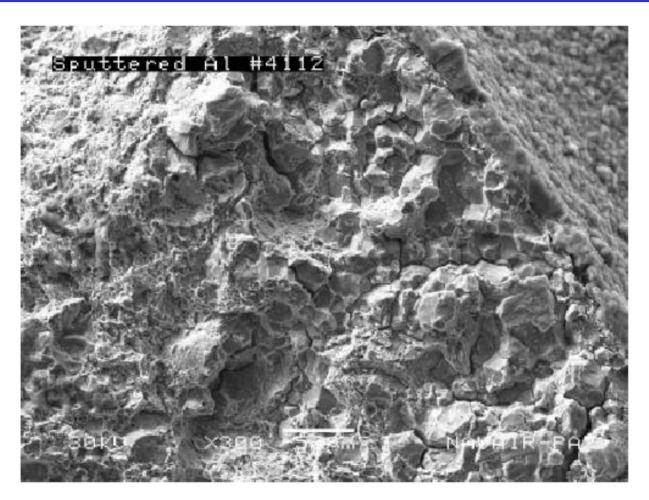


Close-up of IVD Al tested in DI water.

- Intergranular fracture, clear grain boundary separation.
- Coating appears at the top of the cross section.

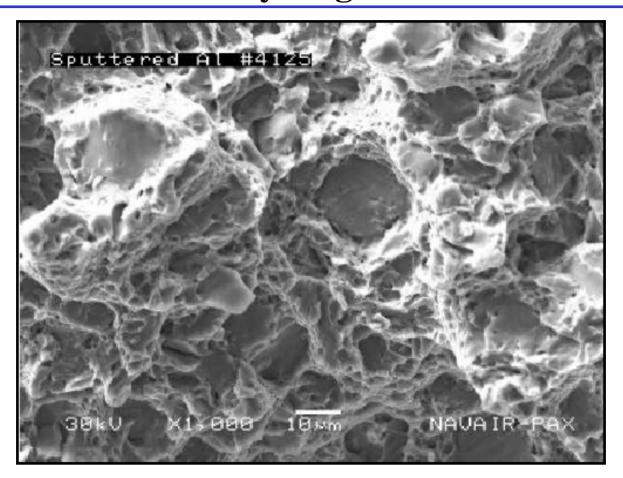


Sputtered Aluminum tested in seawater, failed at 55.5% NFS @ 25.7 hrs.



Close-up of Sputtered Aluminum tested in seawater.

Shows intergranular fracture at surface of specimen.



Close-up of Sputtered Aluminum tested in seawater.

• Shows overload area with mixed ductile dimples and smooth facets suggestive of brittle fracture.

Test Results JTP Section 6.1 Hydrogen Re-Embrittlement / Stress Corrosion Cracking

- Acceptance Criteria: Not established, could be based on time to failure for the cadmium plated test specimens.
- Specimens: ASTM F 519 Type 1d, 4340 alloy steel, HRC 51-53.
- Loading profile: 65% notched bend fracture load, GM9540P cyclic corrosion test to failure.
- Best performance:
 - Alumiplate outperformed all coatings by a large margin, including Cd.
 - Test too severe to discriminate performance.
- Test conducted at Army Research Laboratory.

Test Results JTP Section 6.1 Hydrogen Re-Embrittlement / Stress Corrosion Cracking

Coating	<u>Time to</u> <u>Failure</u>	<u>Cycles</u> <u>Completed</u>	<u>Rank</u>	
Alumiplate	510 hrs.	23.9	1	
Sputtered AI	396 mins.	0.3	2	
LHE Cd	250 mins.	0.2	3	
IVD Aluminum	78 mins.	0.06	0.06 4	
ZnNi (Boeing acidic)	62 mins.	0.05	5	
LHE ZnNi (Dipsol IZ-C17)	47 mins.	0.04	6	

- Acceptance Criteria: No separation (flaking, peeling, or blistering) of the coating from the basis metal. Cracking is acceptable.
- Test Setup: Bend specimen back and forth through 180° until coating and/or substrate ruptures.
- Performance: IVD Aluminum, Brush LHE Cadmium and Sermetel 249/273 had adhesion problems.

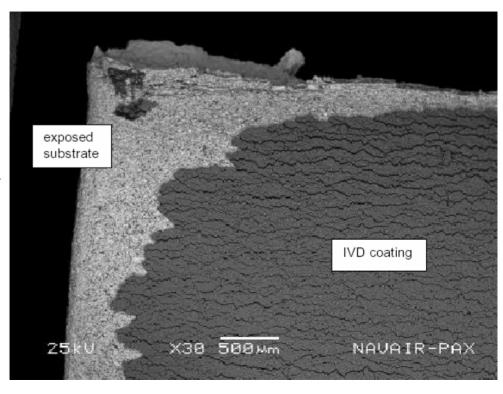


Protective transparency sheet under panel.

Coating	Pass/Fail	Comment
LHE Cadmium	Pass	
IVD Aluminum	Fail	Significant flaking/peeling
Alumiplate	Pass	Very minor peeling at broken edge
Zn-Ni (Boeing, acidic)	Pass	
Sputtered Aluminum	Pass	
Brush LHE Cadmium	Fail	Significant flaking/peeling
Brush Sn-Zn	Pass	
Sermetel 249/273	Fail	Significant flaking/peeling/blistering
Brush Zn-Ni	Pass	
Zn-Ni (Dipsol LHE)	Pass	
Sn-Zn	Pass	

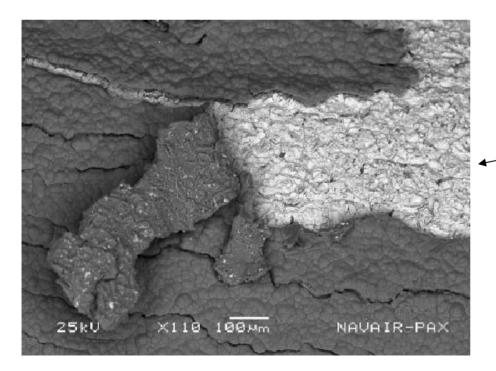
IVD Aluminum coating flaked off of panel.

SEM/EDS analysis confirm exposed substrate.

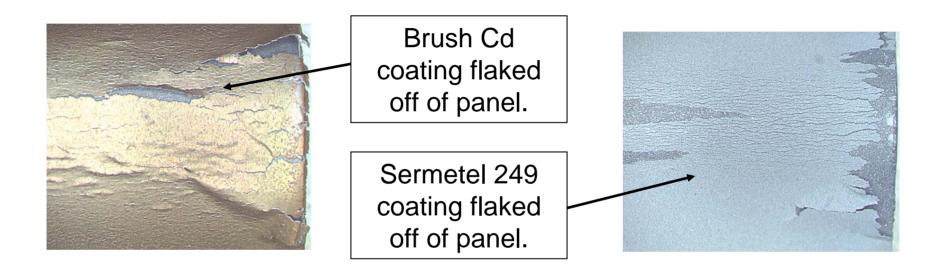


Minor peeling of Alumiplate coating from panel.





Coating peeled back gently with razor.
SEM/EDS analysis confirm exposed substrate.





Brush ZnNi coating cracked but did not pop off panel.

Downselection of Coatings for Phase II effort

Primary alternatives

- Sputtered Aluminum
- Alumiplate
- Dipsol 1Z-C17 LHE ZnNi

Repair alternatives

- Brush ZnNi
- Brush SnZn
- Sermetel 249/273
- Z.R.C. Cold Galvanizing Compound (new Navy nomination)

Voting Results Downselection of Coatings for Phase II effort

Coating	Votes to DROP
Sputtered Aluminum	1
Alumiplate	0
Dipsol 1Z-C17 LHE ZnNi	0
Boeing acidic ZnNi	9
None (primary)	14
Brush ZnNi	7
Brush SnZn	3
Sermetel 249/273	7
None (repair)	4

Voting Results

Downselection of Coatings for Phase II effort

Boeing Acidic ZnNi

 Military vote: 2 of 3 services voted to drop it, 7 votes to drop it vs. 3 to drop no coatings

Brush ZnNi

- None of the military services voted to drop this
- NADEP Jax specifically requested this be retained, will leverage with their dem/val of the process

Sermetel 249/273

- Lack of options for Al repair prompted retaining this
- Poor corrosion and adhesion are a concern

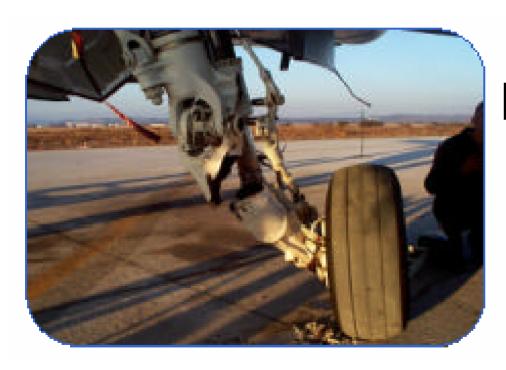
Z.R.C. Cold Galvanizing Compound

- Navy nominates for inclusion, Depot concurrence
- Passed more severe embrittlement test than in JTP

Phase II Tests

Test Category	Test	Testing Facility			
	PHASE II				
	Appearance (JTP 3.1.1)	CTC			
General Properties	Throwing power and alloy composition uniformity (JTP 3.1.2)	CTC			
	Strippability (JTP 3.1.3)	NAVAIR			
	Galvanic potential (JTP 3.1.4)	ARL			
Adhesion	Bend adhesion (JTP 3.2.1)	NAVAIR			
Adicsion	Paint adhesion (JTP 3.2.2)	NAVAIR			
	Unscribed NSS* (bare) (JTP 3.3.1)	ARL			
	Scribed NSS* (bare) (JTP 3.3.2)	ARL			
Corrosion	Galvanic corrosion resistance (3.3.3)	ARL			
Corrosion	Fluid corrosion resistance (3.3.4)	ARL			
	Scribed w/ primer & topcoat (3.3.5)	NAVAIR (paint) ARL (test)			
	SO ₂ salt fog (JTP 4.1)	NAVAIR			
Lubricity	Run-on/Break-away torque (JTP 3.4.1)	WMTR			
Lubricity	Torque-tension (JTP 3.4.2)	WMTR			
	Appearance	CTC			
	Bend adhesion	ARL			
Reparability (JTP 3.7.1)	Paint adhesion	ARL			
	Unscribed corrosion resistance	ARL			
	Scribed corrosion resistance	ARL			
Quality Assurance	Hydrogen embrittlement – notched bar (JTP 3.6.1)	NAVAIR			

What we are trying to prevent...



Landing Gear Stress Corrosion Cracking

...to keep our forces safe and ready.



Backup Info.

Sputtered Aluminum Processing Information:

Coater: Marshall Laboratories

<u>Processing Sequence</u> (all specimen types):

- •Grit blast
- •Isopropyl swab and rinse
- •Glow discharge clean for 15 minutes in 10mTorr argon atmosphere.
- •Plug and coat sputter process. Specimens were mounted radially around the cathode approximately 4" away from cathode surface. Specimens were sputtered for 2.5 hours at about 8kW power, targeting 0.6 to 0.8 mils coating thickness.

Conversion coating applied at NAS Patuxent River Inorganic Coating Laboratory

- Deionized water rinse
- •Conversion coat in Alodine 1200S for 90 seconds at room temperature
- •Tap water rinse
- Deionized water rinse
- •Air dry

Additional evaluation of IVD Aluminum and Alumiplate bend adhesion specimens was conducted to determine if the aluminum coatings had lifted from the substrate steel panel or whether the chromate conversion coating had lifted from the aluminum coating, and the aluminum coating remained adherent to the steel panel. Evaluation indicated the aluminum coatings had lifted from the steel panel in both cases. The Alumiplate coating had only slightly lifted from the panels during the bend adhesion test, and a thin razor was subsequently used to gently peel an already lifted portion of the coating up from the panel to facilitate further evaluation. See attached pdf document titled Cadmium Alternatives Substrate for detailed analysis of the specimens.

In general the coating thickness was measured on the C-ring outer diameters, and the coverage in the notch was observed under at least 40X magnification for the C-rings and notched round bars. Thickness in the notch was not measured.

Coating thicknesses were measured using an Elcometer 456 Coating Thickness Gauge with ferrous F1 probe on the Type 1d C-rings only. Accuracy of the gauge is ±0.1 mil. Six measurements were taken on each specimen along the length of the notch at three equidistant points on each side of the notch approximately ¼" out from the notch. The coating thicknesses documented below are the average of 10 specimens per coating and 6 measurements per specimen.

Coating coverage of 1"x4" bend adhesion panels was good for all coatings. No magnification was used for inspection of the panels.

• Coating: Tin-Zinc Plating

- <u>Coating composition</u>: 75 to 85% tin and 15 to 25% zinc (per coater documentation).
- <u>Coating thickness</u>: 0.4 to 0.5mil (per coater documentation), not measured by NAS Pax River lab.
- Coating coverage: Type 1a.1 specimens were not inspected by NAS Pax River for coverage in the notch prior to HE tests. Eight extra, untested specimens were inspected for coverage in the notch using 100X magnification. All eight specimens appeared to have full coating coverage in the notch, however, the coating surface contained numerous blisters/pits and areas of red rust.

• Coating: Sputtered Aluminum

- <u>Coating composition</u>: 100% aluminum
- <u>Coating thickness</u>: 0.6 to 0.8 mils targeted. 2.15 mils (0.32 standard deviation) actual.
- Coating coverage: Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification prior to HE tests. All specimens appeared to have full coating coverage in the notch. There were light grey spots on four specimens. One specimen had a small scratch on the notch skirt. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

• Coating: Ion Vapor Deposited (IVD) Aluminum (unpeened)

- <u>Coating composition</u>: 100% aluminum
- <u>Coating thickness (round bars)</u>: Class 2 minimum (0.5 mil) targeted. Not measured.
- <u>Coating coverage (round bars)</u>: Notch examined by coater under 10X magnification for full coverage all specimens showed full coverage in the notch. Specimens were inspected by NAS Pax River for coverage in the notch using 100X magnification after HE tests conducted. All specimens appeared to have full coating coverage in the notch.
- <u>Coating thickness (C-rings)</u>: Class 2 minimum (0.5 mil) targeted. 1.53 mils (0.18 standard deviation) measured.
- Coating coverage (C-rings): Notch examined by coater under 10X magnification prior to conversion coating step coverage appeared complete. Notch examined by coater under 10X magnification for full coverage. Four specimens showed full coverage into the root of the notch indicated by uniform conversion coating color. Six specimens showed non-uniform color in the root of the notch leading to questions about the notch coverage. Specimens reexamined at 30X. Believe the notch to be completely coated as evidenced by the lack of any corrosion in the notch and the examination prior to conversion coat. These six specimens were identified by a question mark on each bag in which they were stored. Specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.
- <u>Coating thickness (panels)</u>: Class 2 minimum (0.5 mil) targeted. Not measured.
- <u>Coater notation (panels)</u>: One of the specimens was lost. A second set was prepared from Hill AFB stock (4130 steel), and labeled "Spare Adhesion, IVD Aluminum". The three specimens from the original set was used for the adhesion test.

• Coating: Low Hydrogen Embrittlement (LHE) Cadmium

- Coating composition: 100% cadmium
- <u>Coating thickness (round bars)</u>: No attempt was made to measure coating thickness. Using cathode efficiency of 75% (which is typical of this solution at the current density used) 8.0 minutes will result in a coating weight equivalent to 0.6mil thick. Due to the porosity of the deposit, the actual deposit may be thicker than this.
- Coating coverage (round bars): Notch examined by coater under 10X magnification for full coverage. Two of the 18 specimens showed spots that looked like bare spots (no cadmium coating). Those two specimens were identified by the coater with blue paper over the tissue wrap. Those two specimens were not used for testing. Specimens were inspected by NAS Pax River for coverage in the notch using 100X magnification after HE tests conducted. In general specimens appeared to have full coating coverage in the notch, on some specimens the coverage appeared slightly light to bare.
- <u>Coating thickness (C-ring)</u>: 0.51 mils (0.10 standard deviation) measured.
- Coating coverage (C-rings): Notch examined by coater under 10X magnification prior to bake step all specimens appeared to have full coverage in the notch. Notch examined by coater under 10X magnification after conversion coating step for full coverage. All specimens showed non-uniform color in the root of the notch leading to questions about the notch coverage. Specimens re-examined under 30X magnification. Believe the notch to be completely coated as evidenced by the lack of any corrosion in the notch and the examination prior to conversion coat. Specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.
- <u>Coating thickness (panels)</u>: No attempt was made to measure coating thickness. Eight minutes plating time is equivalent to 0.6mil thick on average. However, it appeared that there was considerable edge effect likely resulting in heavier deposit around the outside edges.

• Coating: Electroplated Aluminum (Alumiplate)

- Coating composition: 100% aluminum (no underplate)
- <u>Coating thickness</u>: Class 2 (minimum 0.5 mil) certified. 0.92 mils targeted. 1.08 mils average calculated based on coating weight. 1.41 mils (0.25 standard deviation) measured by Pax River.
- <u>Coating coverage</u>: Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 100X magnification after HE tests conducted. All specimens appeared to have full coating coverage in the notch. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

• Coating: Zinc-Nickel, Boeing Acidic

- Coating composition: 90.5 to 91.5% zinc, 8.5 to 9.5% nickel
- Coating thickness: 0.5 mil targeted. 0.29 mils (0.06 standard deviation) actual.
- Coating coverage: Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 100X magnification after HE tests conducted. All specimens appeared to have full coating coverage in the notch. One specimen that was tested in the 200-hr HE test had a small chip in the coating on the notch skirt. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

• Coating: Low Hydrogen Embrittlement Zinc-Nickel, Dispsol IZ-C17

- Coating composition: 12 to 16% nickel, balance zinc (per Dipsol)
- Coating thickness: 0.5 mils targeted. 0.53 mils (0.11 standard deviation) measured.
- <u>Coating coverage</u>: Coater plated the reduced gage section of Type 1a.1 (no plating on threaded ends) –
 Visual inspection of notch showed that entire notch was plated. Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification prior to HE tests. All specimens appeared to have full coating coverage in the notch. Type 1d specimens were inspected by NAS Pax River for coverage in the notch using 3.5X magnification. All specimens appeared to have full coating coverage in the notch.

• Coating: Brush Cadmium Plating, SIFCO 2023

- Coating composition: 100% cadmium
- *Coating thickness*: 0.5 mil targeted. Not measured.
- Coating coverage: Per the coater, cadmium plating appeared to be uniform and the notch on the Type 1a.1 specimens appeared to also have good plating coverage, except for #AL4068 (had some flaking in the notch most likely due to poor surface prep). Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification after HE tests were conducted. All specimens appeared to have full coating coverage in the notch. Untested specimens were also inspected at 40X magnification. Two of four untested specimens had exposed (uncoated) area in the notch root and the coating looked thin around the entire notch root of one specimen.
- <u>Coater notation</u>: Type 1a.1 specimens were inspected and cloth threads were seen on the plating. These were cleaned with scotch brite pads and then conversion coated for another 15 seconds.

• Coating: Brush Zinc-Nickel Plating, SIFCO 4018

- <u>Coating composition</u>: 8 to 12% nickel, balance zinc (per SIFCO)
- Coating thickness: 0.5 mil targeted. Not measured.
- <u>Coating coverage</u>: Per the coater, Zn-Ni plating did not have a uniform appearance on some (8 of 18) of the notches on the Type 1a.1 specimens and are noted in documentation that accompanied the test specimens. The remainder of the specimens (10 of 18) appeared to have good plating coverage. Type 1a.1 specimens were inspected by NAS Pax River for coverage in the notch using 40X magnification prior to HE tests. All specimens except one had a shiny notch root possibly indicating thin coating. Three specimens appeared to have bare spots in the notch root. Only the specimens indicated as having good coating coverage by the coater were used for testing.

• Coating: Brush Tin-Zinc Plating, LDC 5030

- <u>Coating composition</u>: 70 to 75% tin, balance zinc (per LDC)
- <u>Coating thickness</u>: 0.5 mil targeted. Not measured.
- <u>Coating coverage</u>: Per the coater, Sn-Zn plating appeared to be uniform and the notch on the Type 1a.1 specimens appeared to also have good plating coverage.

• Coating: Spray-on Sermetel 249/273

- <u>Coating composition</u>: The coating contains aluminum and zinc powder in an inorganic binder system (per Sermatech)
- *Coating thickness*: 1.0 to 2.0 mil targeted. Not measured.
- <u>Coating coverage</u>: Coater spray coated the reduced gage section of Type 1a.1 (no coating on threaded ends) Visual inspection of notch showed that entire notch was coated. Type 1a.1 specimens were not inspected by NAS Pax River for coverage in the notch prior to HE tests. Extra, untested specimens were inspected for coverage in the notch using 40X magnification. All specimens appeared to have full coating coverage in the notch.